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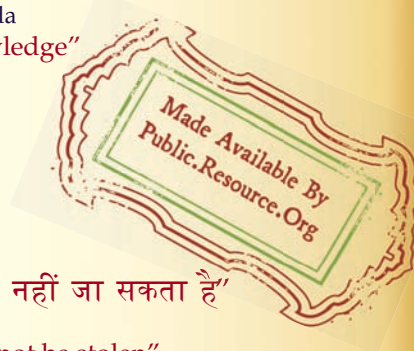
IS 12491 (1988): Method for determination of magnesium by atomic absorption spectrophotometry [CHD 1: Inorganic Chemicals]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

**METHOD FOR DETERMINATION OF
MAGNESIUM BY ATOMIC ABSORPTION
SPECTROPHOTOMETRY**

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Indian Standard

METHOD FOR DETERMINATION OF MAGNESIUM BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 31 May 1988, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

0.2 The atomic absorption spectrophotometric method is based on the fact that metallic atoms present in ground state will absorb incident radiation from the source (normally hollow cathode lamp) of the same wavelength as they emit when excited. When radiation from the given excited element is passed through the flame containing ground state atoms of that element, the intensity of transmitted radiation will decrease in proportion to the amount of ground state atoms present in the flame. A hollow cathode lamp with cathode made of the element to be determined, or a suitable electrode discharge lamp provides the radiation. The metal atoms to be measured are placed in the beam of radiation by aspirating the sample

solution into the flame. A monochromator isolates the characteristic radiation from the hollow cathode lamp and the photosensitive device measures the attenuated transmitted radiation. The absorbance is taken as the measure of concentration of the analyzed element. Various metallic elements including magnesium may be precisely analyzed by this technique.

0.3 Various products where magnesium estimation may be required, include metals and alloys, minerals and ores, slags and other refractory oxides, coal, coke and crude oil, atmospheric particulates, etc.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

*Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard prescribes the method for determination of magnesium by atomic absorption spectrophotometry.

2. OUTLINE OF THE METHOD

2.1 The sample is brought into solution by suitable treatment with acid combinations, diluted with water, filtered, and suitable dilutions are made for aspiration into the air-acetylene flame. The standard solution is made in the same way for calibration. The most sensitive magnesium line is 285.2 nm; however, other line suitable for higher concentrations can also be used.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and deionized water shall be used.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. INTERFERENCE

4.1 Aluminium and silicon depress the response in air-acetylene flame. This effect may be reduced by the addition of 0.2 percent lanthanum chloride or strontium chloride to both samples and calibration solutions, or by the use of the hotter flame. For determinations in nitrous oxide-acetylene flame, an ionization buffer, such as 0.1 percent (m/v) of potassium chloride, shall be added to all solutions. The presence of alkali metals will depress the response at this wavelength and so the calibration solutions must be matched to concentrations in the sample solutions. It has been reported that phosphate may depress the response in the air-acetylene flame. The effect may be overcome by the addition of releasing agents or the use of the hotter flame (nitrous oxide-acetylene) as described for the presence of aluminium and silicon.

5. APPARATUS

5.1 Atomic absorption spectrophotometer provided with background corrector and having the following parameters:

5.1.1 Lamp Current — depending on the lamp and the instrument used.

5.1.2 Fuel — air-acetylene or nitrous oxide-acetylene flame.

5.1.3 Flame Stoichiometry — oxidizing.

5.1.4 Slit Adjustment — depending on the instrument

5.1.5 Wavelength and Working Range:

Wavelength (nm)	Working Range ($\mu\text{g/ml}$)
285.2	0.005 to 0.10
202.6	0.2 to 4.0

6. REAGENTS

6.1 Pure Magnesium Metal — 99.99 percent.

6.2 Concentrated Hydrochloric Acid — See IS : 265-1976*.

6.3 Standard Magnesium Solution — Dissolve 1.0 g of magnesium metal in 50 ml of 5 N hydrochloric acid. Dilute to 1 litre in a volumetric flask with water. Store in a polyethylene bottle.

7. SAMPLE PREPARATION

7.1 Metals and Alloys — A suitable quantity of sample is dissolved in hydrochloric acid or mixture of hydrochloric acid and nitric acid, evaporated to dryness, again dissolved in hydrochloric acid, diluted, filtered and made up to known volume. A suitable dilution is made for determination of magnesium before aspirating in the flame.

7.2 Water and Effluents — Magnesium content in water is usually very high. Suitable dilution is required before feeding to flame. During each dilution, one or two millilitres of hydrochloric acid is to be added.

7.3 Minerals and Ores — Finely divided particles are dissolved in sulphuric acid (5 ml) and hydrofluoric acid (10 ml) mixture in a platinum crucible and evaporated to dryness. It is taken again in 5 ml sulphuric acid (10 percent) and heated to dryness. The residue is treated with hydrochloric acid (1 ml) and water. It is then heated on a water-bath till it dissolves. The solution is made up to known volume.

*Specification for hydrochloric acid (second revision).

7.4 Slags and Other Refractory Oxides — Finely divided particles, nearly 0.5 g, are dissolved in hydrochloric acid (15 ml) and nitric acid (1 ml) in a crucible/dish made of platinum or polytetrafluoroethylene (PTFE) or equivalent resistant material, heated and evaporated to dryness. Then hydrofluoric acid (10 ml) and sulphuric acid (1 ml) are added, heated to fumes and then to dryness. It is then cooled and the mass is taken in hydrochloric acid. Filtered, and if there is any residue, it is to be ignited, fused with sodium carbonate, taken in hydrochloric acid and mixed with the original filtrate. Suitable dilution with 0.2 percent lanthanum chloride or strontium chloride solution is required before feeding to flame or use hotter flame if lanthanum chloride or strontium chloride is not available.

7.5 Coal Coke and Crude Oil — After ashing at 550°C, it is dissolved in *aqua regia*, filtered and diluted to volume. In case of crude oil, charring should be done under infrared lamp before ashing.

7.6 Atmospheric Particulates — Membrane filter containing particulates is leached in dilute nitric acid and filtered. The solution is made to known volume with water.

8. PROCEDURE

8.1 Optimize the response of the instrument by the adjustment of burner height and flame. Aspirate to get zero absorption, when stable response is observed, aspirate standard at least 4 times and note down the absorption. Aspirate the sample to get the absorption of the sample, prepare the calibration curve by plotting the net absorption value of the standard against concentration in $\mu\text{g/ml}$ of magnesium. Locate the point of the sample absorption and calculate the concentration of magnesium in the sample.

NOTE — If the hotter flame is used, addition of lanthanum chloride or strontium chloride solution to the standard and the sample solution may be omitted.

9. CALCULATION

Magnesium, percent by mass

$$= \frac{C \times V}{10^6} \times \frac{100}{M}$$

where

C = concentration of magnesium in $\mu\text{g/ml}$ in final solution,

V = volume in ml of final solution, and

M = mass in g of the sample in final solution.